FATIGUE BEHAVIOUR OF FLAX REINFORCED COMPOSITES

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Abstract

The present study focuses on the characterization and evaluation of fatigue behaviour of flax-epoxy composites. A better understanding of this behaviour allows the prediction of long-term properties to assess the viability and long-term durability of these materials. The purpose of this work is to systematically study the tension-tension fatigue behaviour of flax fibre composites for five textile architectures and four laminate configurations, which are used in a wide range of applications. The fibre architecture was found to have a strong effect on the fatigue behaviour where higher strength and modulus combinations have a delayed damage initiation and increased fatigue life as well as a reduced damage propagation rate and higher energy dissipation in the early stages.

1. Introduction

The use of flax fibre in the composites industry is increasing, thanks to their advantageous characteristics such as acoustic and thermal insulation, vibration damping, as well as renewability. In terms of mechanical performance, flax is one of the strongest of the natural fibre family and is known to be as stiff as glass fibre. As of today, a major lack of data on flax fibre durability properties, such as fatigue behaviour, has been limiting the use of these fibres in high performance applications like wind turbine blades and sports goods. Very few papers related to the fatigue behaviour investigation were published such as the ones by Liang et al.[1] on flax/epoxy composites, Shah et al.[2] on flax/polyester composites and Towo et al.[3] on sisal/epoxy composites. Liang et al. [1] have found out that glass fibre- reinforced epoxy composites have an increased resistance to fatigue loading compared to flax epoxy composites which is due to their higher static strength (103 vs 79 MPa). On the other hand, glass composites have major reduction in fatigue life illustrated by a steeper S-N curve with a drop of stress in comparison to flax/epoxy (21 MPa/decade vs 7 MPa/decade) showing a stable fatigue behaviour through time for the flax/epoxy composite. Silva et al. [4] have shown for sisal fibres, that at fatigue stresses below 50% of the ultimate tensile strength (UTS), all tested fibres can resist more than 10^6 cycles as well as showing a slight increase of the stiffness in the early cycles. A study published in 2014 by Shahzad et al. [5] showed that hemp fibres-reinforced composites were less fatigue sensitive than their glass fibre counterpart in tension-tension fatigue which could be due to the lower stiffness degradation at equal normalized stress levels (S/S_0). As it is the case for all properties characterization, the textile architecture, fibre and matrix types and fibre volume content were found to have a strong effect on the fatigue life. It is expected that higher static properties are a sign of superior fatigue loading capacities [6]. It was shown by Gassan et al. [7] that higher fibre strength and modulus natural fibre-based composites, which have a better fibre–matrix adhesion, have a delayed damage initiation and reduced damage propagation. Furthermore, Shah et al. [6] found that the fatigue strength degradation rates, obtained from the slope of the S/N curve of flax/polyester composites, are lower than for their glass counterparts. Throughout the lifetime of composites, they will encounter certain damage that will affect their overall performance. Although fatigue loading does not instantly reduce the strength of the composite, it does have an effect on the stiffness. During testing, early damage causes a fast and steep decrease of the stiffness which is followed by a stabilisation and slow degradation of the composite properties. Close to failure, a strong decrease in stiffness in heavily damaged zones will cause the material to break [8, 9]. To monitor the damage, the cyclic hysteresis is used by many authors in order to evaluate the loss of stiffness, material energy dissipation and damage development [3, 10]. The aim of this study is to investigate *S*–*N* diagrams constructed from fatigue data at stress ratios of $R = 0.1$ (tension–tension) at various stress levels and this for nine different flax textile architectures and laminates combined with an epoxy matrix. The composites were manufactured via the resin transfer moulding process, mechanically tested and their performance compared. Strain monitoring was used during testing in order to assess the stiffness degradation rates as well as the hysteresis loops to investigate the energy dissipation efficiency of each material. This investigation is crucial in order to assess the durability of this type of materials since these properties have a great influence on the service life, product safety and liability.

2. Experimental

2.1 Materials

The flax fibre reinforced composites were made by combining a thermoset epoxy matrix with nine flax textile configurations. For the cross-linking, the matrix *Epikote 828LVEL* (η ≈ 10-12 Pa.s) is mixed with the *Dytek DCH-99* hardener at a 15.2 phr ratio. The 2 mm thick laminates are composed of several layers of fabrics in order to obtain a fibre volume fraction (Vf) of ≈ 40% except for the random mat laminate which has a Vf = 30%. The different textile and matrix properties are described in Table1.

Flax fibre preform architectures	**Mat**	**Plain weave**	**Twill**			**Quasi-UD***	**Unidirectional**
Areal density ρ_{sur} (g/m^2)	300	285	400	200	150	300	200
1 ply thickness (mm)	0.21	0.2	0.23	0.14	0.103	0.21	0.14
Acronym	Random Mat	Plain Weave	Low Twist Twill	Medium Twist Twill	High Twist Twill	Quasi-UD	UD

Matrix Type	**Commercial Name**	**Modulus (GPa)**	**Strength (MPa)**
Epoxy (Thermoset)	Epikote 828 LVEL	2.7	70

* 90% of fibres in 0° direction and 10% in the 90° direction.

Table 1: Flax fibres and epoxy matrix characteristics.

For the Quasi-UD and UD laminate, both an UD configuration and a symmetric cross-ply lay-up were used $(0,90)_{xs}$, where x is the number of layers needed to achieve a Vf=40% using the following Equation 1:

$$Vf = \frac{\rho_{surf} * N}{\rho_{vol} * h}$$ **Equation 1**

Where ρ_{surf} is the aerial density (gsm) and ρ_{vol} is the density of the fibers (1.45g/cm³), *N* is the number of layers used and *h* is the composite laminate thickness (2mm).

2.2 Composite manufacturing and Testing

The composites were manufactured using the Resin Transfer Molding (RTM) technique. The fibres were placed between the two rigid steel mold parts and were clamped together with an hydraulic press. A spacer was added to adjust the depth of the cavity in order to obtain the thickness of 2mm± 0.05mm. The experimental parameters were P_{inlet}=1bar, P_{out}=-1bar and a consolidation pressure of 3 bars. The injection temperature was 40°C and then increased to 70°C for 1h after the injection to cure the part. Later on, the plates were post-cured at 150°C for 1h.

Tensile mechanical tests were performed using an Instron #4505 testing machine in the longitudinal direction. Samples of 2x25x250 mm were tested using a crosshead speed of 2 mm/min, a load cell of 30 kN and a gauge length of 150 mm according to ASTM D3039 standard. A 50mm gauge length extensometer was used to monitor the strain. A minimum of 5 samples, previously end-tabbed to avoid stress concentration at the grips, were tested to ensure reproducibility and reliable standard deviations. With these tests the Ultimate Tensile Strength (UTS), see Figure 1, is determined which is used to determine the load level used during fatigue testing. Tension–tension fatigue tests were done following the ASTM D3479 standard on a MTS hydraulic fatigue testing device with a 100kN load cell. The loading ratio was set to R= 0.1 with a loading frequency of 5 Hz and load levels (S/S_o) ranging from 0.3 to 0.9 UTS. The tests were conducted at 22°C using UTS data from Figure 1. Samples were prepared with the same dimensions as for static tensile testing and end-tabbed.. The test is set to stop if the samples do not fail after 10E6 cycles, referred to as N_{max}. Hysteresis loops and stiffness degradation were captured continuously using a fatigue rated extensometer with a gauge length of 50 mm. A minimum of 4 samples for each load level were tested.

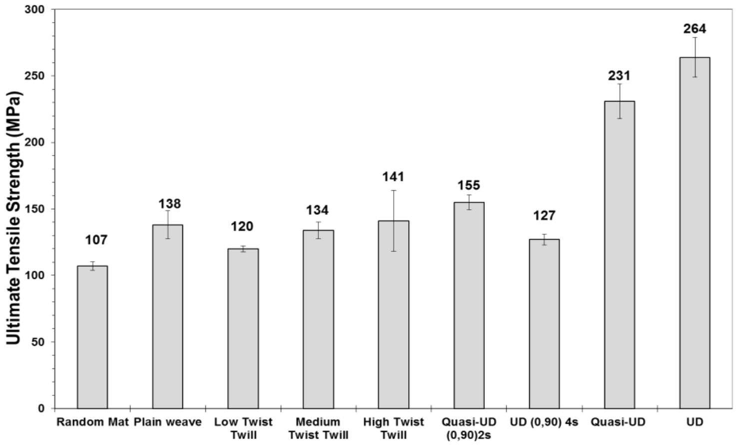

Figure 1: Ultimate Tensile Strength Results for various Flax/Epoxy composites.

3. Results and Discussion

3.1 Fatigue life assessment

Tensile–tensile fatigue cycling was carried out at load levels corresponding to various fractions of the ultimate tensile strength (UTS) registered during quasi-static testing. This value is referred to as S_0, and is later used to normalize the load levels (S/S_o) that the samples are subjected to. Although there is scatter in the flax fibre composite fatigue data, such scatter is fairly common place in fatigue lifetime data for composites and it has previously been reported for synthetic fibre-reinforced composites where the normal scatter in static strength data can be increased by random damage during fatigue loading [11]. In Figure 4, showing the fatigue life of the nine chosen flax/epoxy configurations vs the maximum tensile stress (*S*–N), it can be seen that the random mat is the lowest performing laminate compared to textiles, cross-ply and UD laminates, as expected. Furthermore, the slopes of the *S*–N curves show a slightly steeper decrease for the Quasi-UD composite in comparison to the UD composite while all the textiles (plain weave, twills) and the cross-ply laminates display similar curves (see *Obtained **from average of four fatigue tested samples**

Table 2). This observation is consistent with the static tests data meaning that composites displaying high static strength have a longer lifetime. However, higher static strength becomes less critical as the number of fatigue cycles imposed increases as Towo et al. [3] stated for sisal fibre composites. Furthermore, a steeper slope is a sign of a high fatigue degradation rate during cyclic loading. As the loading level decreases, all textile and cross-ply laminates are achieving comparable lifetimes which suggests that at lower stress levels, the need for a high quality textile may become unnecessary. Moreover, if the UD composites (UTS= 264 MPa) is compared to the UD (0,90) cross-ply configuration (UTS=127 MPa), it is possible to observe that the at high cyclic loading, the cross ply tends present an improved plateau-like behaviour while the UD tends to degrade in a linear fashion due to the increased off-axis loading angle even though the degradation rate is faster for a UD (0,90)4s than the UD samples, as also found by Shah et al. [6] on Polyester-flax composites. Nevertheless, during normal operation, some components are subjected to static loads rather than fatigue loads hence the importance of the wise choice of architecture that can increase the load carrying capacity at low load level [3]. The power–law regression curves, as seen in ***Obtained from average of four fatigue tested samples**

Table 2 and Figure 4, are following quite well the experimental data (excluding the static strength values) with an $R^2 > 0.8$.

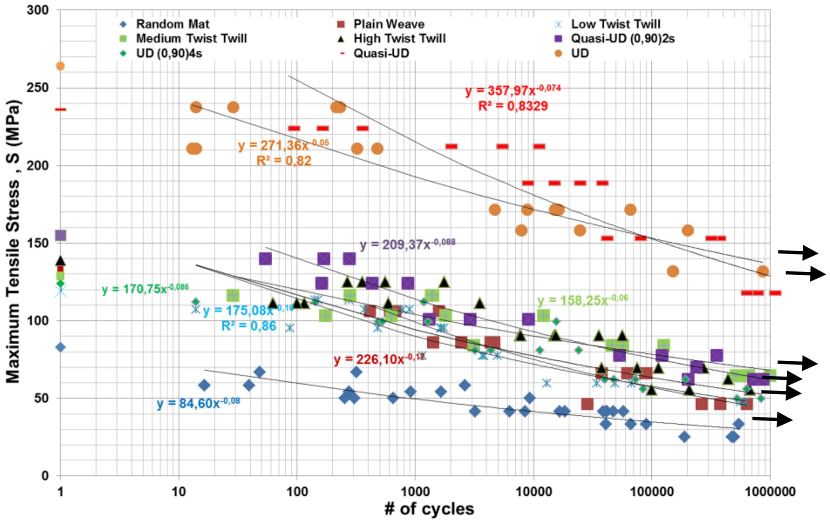

Figure 2: Stress-life data for untreated flax-epoxy composites at *R* = 0.1 (including static strengths).

	Fatigue life power law curves	Expected Stress value at 10E6 cycles (MPa)	Experimental Stress value at 10E6 cycles (MPa)*
Random Mat	$84{,}41x^{-0{,}08}$	28	25
Plain Weave	$226{,}10x^{-0{,}12}$	43	46
Low Twist Twill	$175{,}08x^{-0{,}10}$	44	48
Medium Twist Twill	$158{,}25x^{-0{,}06}$	69	65
High Twist Twill	$193{,}37x^{-0{,}086}$	59	56
Quasi-UD (0,90)$_{2S}$	$209{,}37x^{-0{,}088}$	62	62

UD (0,90)$_{4S}$	$170{,}75x^{-0{,}086}$	52	50
Quasi-UD	$357{,}97x^{-0{,}074}$	129	118
UD	$271{,}36x^{-0{,}05}$	136	132

***Obtained from average of four fatigue tested samples**

Table 2: Fatigue life curves for all configurations; expected value at 10E6 cycles.

As the cyclic loading decreases from 0.9 to 0.4, the high initial strength is less dominant, i.e. that it has less influence on the fatigue life, as observed in Figure 2 for Quasi-UD $(0,90)_{2S}$ vs the Low Twist Twill and the UD versus the Quasi-UD which performance almost coincides at 10E5 cycles . The fatigue life prediction, following an assumed power law behaviour, shows an endurance limit (10E6 cycles) of about 28 MPa for the random mat and 43 MPa for plain weave composite, which correlates well with the experimental data which give 25MPa and 46MPa respectively as shown by arrowheads in figure 2 (i.e. non-broken samples). Hence, a peak stress level of 28 MPa (30% of UTS) can be taken as a safe value for the endurance limit at 10E6 cycles for the random mat composite. A similar observation is made for the other architectures. To compare the fatigue sensitivity of the studied materials, the normalised stress (S/S_{o}) versus cycles to failure (N) curve method is used (see Figure 3). Although a clear pattern can be observed for the S/N curves, once these values are normalized to the static strength (S/S_o vs N), no relative difference between all architectures at low load levels (S/S_o<0.5) can be seen besides for the random mat as expected. The normalized curves show that the fatigue strength of the composites at high load level (S/S_o=0.9 and 0.8) is very scattered varying from 100 to 10000 cycles depending on the architecture. For higher load levels, the fatigue strength steadily decreases by an average of 5-10% per decade of cycles from the static strength. A similar value of static strength reduction was reported by Mandell [12] and Shazad [5] for UD E-glass fibre and randomly oriented hemp fibre reinforced composites respectively which means that natural fibres such as hemp and flax demonstrate comparable fatigue sensitivity to unidirectional glass fibre composites [13].

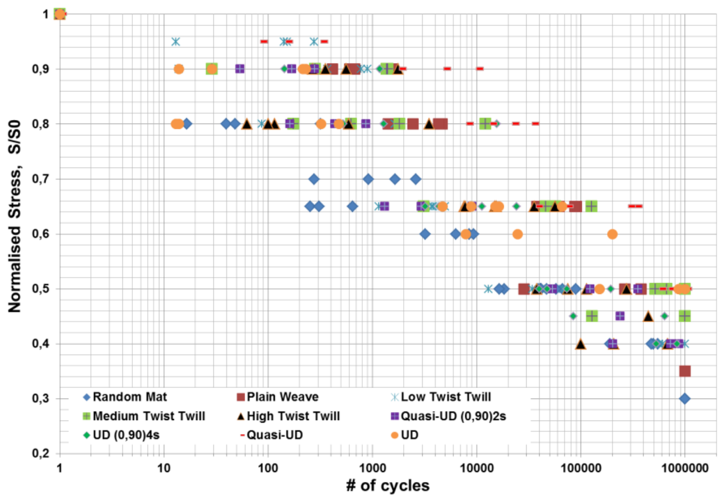

Figure 3: Normalized fatigue data for flax-epoxy composites at *R* = 0.1.

The normalized stress/density-number of cycles to failure (*S/ρ–N*) curves, seen in Figure 4 with ρ=2.55 for glass and ρ=1.45 for flax, show lower fatigue life for flax random mat vs glass random and somewhat equivalent fatigue life for the flax plain woven fabric compared to its glass counterpart tested by Hussain et al. [14]. Flax PW has an enhanced fatigue performance compared to glass PW with the first being able to sustain around 100 000 cycles and the second only 20 000 cycles at 65% of the load/density level. Furthermore, a fatigue investigation by Thwe et al. [15] on bamboo-polypropylene and bamboo–glass polypropylene hybrid composites has demonstrated that the first has an enhanced fatigue resistance compared to the hybrid. This opens the possibility to the use of fully natural fibre based composites in certain types of composite applications.

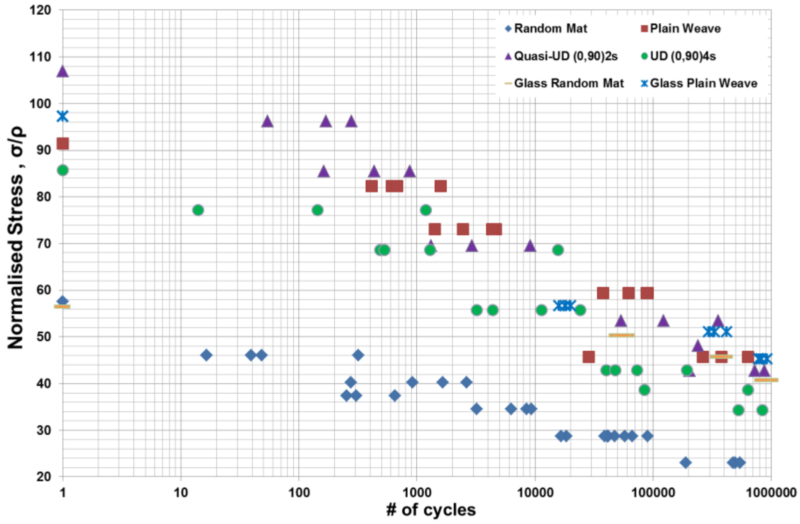

Figure 4: Normalized *S–N* curves according to material density.

3.2 Damage evolution and energy dissipation

The influence of textile architecture was investigated in order to assess the damage initiation and propagation and to determine its influence on the stiffness. Two main parameters were studied: stiffness variation through time and the dissipated energy. The E-modulus was calculated at each cycle by using the slope between the highest and lowest value of the cycle hysteresis. It was observed that flax/epoxy composites with higher strength and modulus properties had a delayed damage initiation as well as a reduced damage propagation rate illustrated by the limited hysteresis (smaller loop area) and their low strain shift (low plastic deformation) as seen in Figure 6. At a 0.5 load level, the stiffness degradation observed is very small. After a slight decrease in the early cycles, the stiffness stays stable. It was found by Vallons et al. [16] that at low load level, the damage in carbon-epoxy cross-ply fabrics happens mainly in the matrix and since the composites' behaviour is mainly fibre-dominated, a rather low decrease in stiffness can be observed.

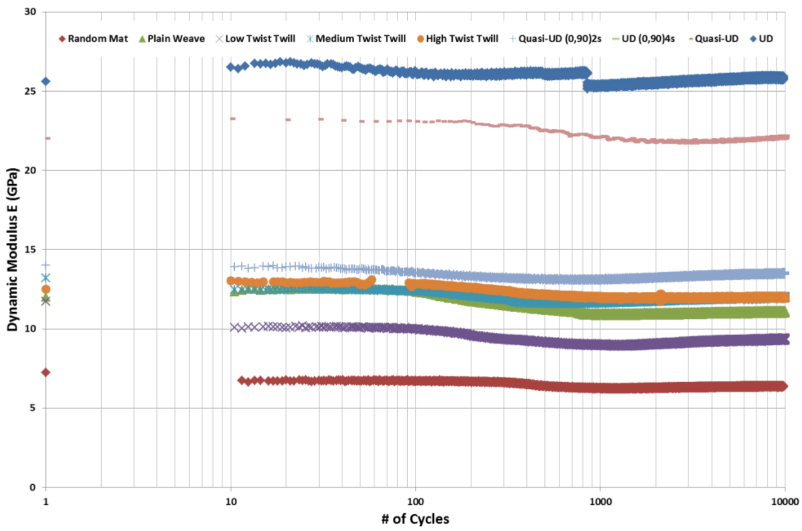

Figure 5: Stiffness degradation curves at S/S0=0,5.

During the fatigue testing, the deformation of and creation of damage in flax/epoxy specimens will dissipate a certain amount of energy during the increasing load phase and only part of this energy is given back during unloading. This phenomenon will leave the specimen with a residual strain that will accumulate through time and affect the overall mechanical properties (↓ of the stiffness) of the composites as well as further defect propagation [17, 18]. This can be seen in Figure 6 where the shift of the hysteresis loop toward increasing strain levels is an indication of permanent plastic deformation (elongation) of the specimen [16]. Additionally, damage development, characterized using stress-strain measurement during cycling, showed a larger stress-strain (σ-ε) hysteresis loop area and an increase of the slope in the early cycles compared to cycles close to the failure of the sample for four of the nine architectures (see Figure 6). The latter behaviour is indicating an increase of the dynamic modulus due to the potential reorientation from 10^o to 0^o of the flax microfibrils present in the elementary fibres in the early cycles [1, 3]. For the random mat, a lot of energy is still being dissipated after 15000 cycles compared to UD or the medium twist twill where the loops are getting slimmer which is a sign that no more damage is created, i.e. the number of matrix cracks reaches a saturation state[16]. For the random mat, since the fibres are in all directions, it is believed that their stiffening is gradual rather than fast as it happens with the pure UD where all the fibres are loaded in the longitudinal direction. For this reason significant hysteresis loops are observed above the level of the other architectures. The wide hysteresis loop in tension-tension fatigue may also be a sign of high damage activity during the initial phase of fatigue loading indicating greater energy dissipation at the fibre–matrix interface [7]. Furthermore, the hysteresis loops captured beyond 1000 cycles show a slight increase in slope which is a sign of a potential increase in the dynamic modulus as a result of stiffening of the composite along the fibre direction.

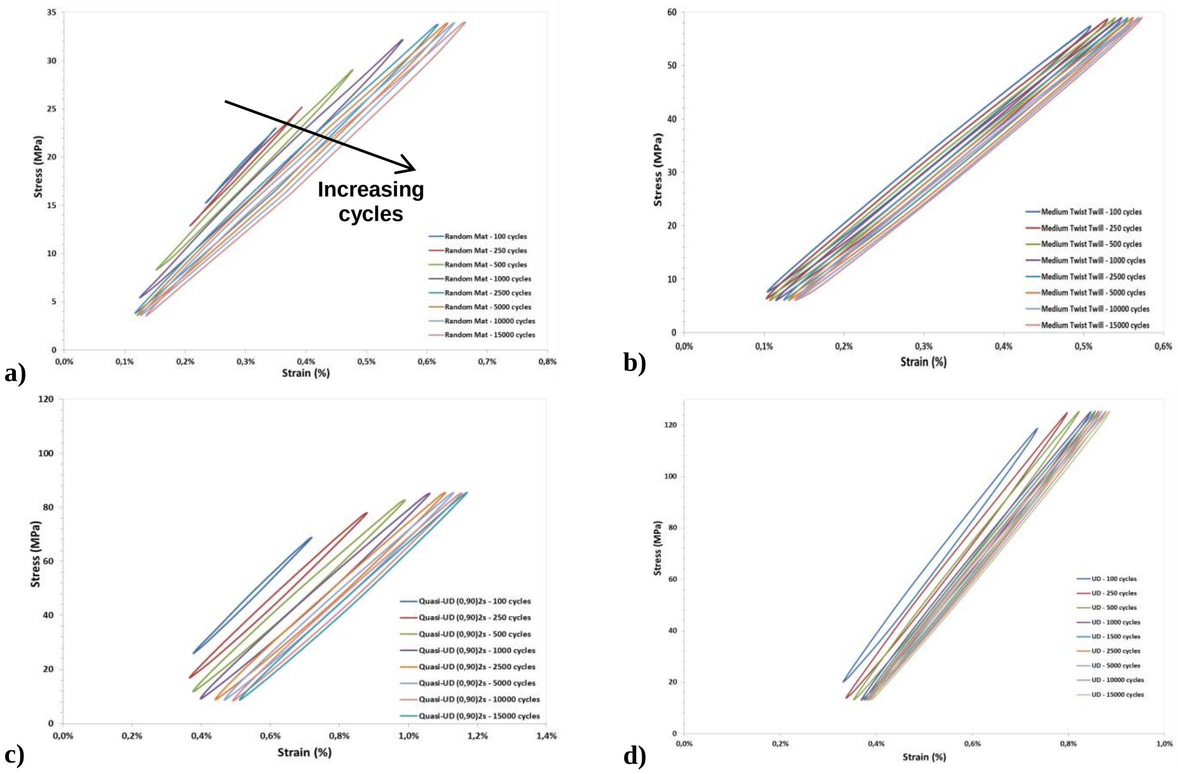

Figure 6: Stress-strain data from fatigue testing for the first 15000 cycles for the a)Random mat, b) Medium Twist Twill, c) Quasi-UD (0,90)2s and d) UD composites at S/S0=0.5.

4. Conclusions

Tension-Tension fatigue tests were carried out in order to assess the long term behaviour of flax-based composites. It was observed that at normalized stress/density, the fatigue life of flax/epoxy PW composite is higher than in case of glass PW composite. As well, the results show that the various architectures have different behaviour, but do not necessarily influence the fatigue lives at lower stress levels and no clear difference could be observed for the woven fabric and cross-ply laminate. Furthermore, the composites dissipate most energy during the initial phase of cyclic loading as shown by the analysis of hysteresis loops in tension–tension. The slight increase in the early cycles of the modulus degradation curves is consistent with stiffening of composites in the fibre direction, as shown by the slight increase in steepness of the slope of the loading side of the hysteresis loop. Overall, the behaviour of flax-epoxy composites is rather similar to synthetic fibre reinforced composites and suitable for many new or existing industrial applications. Further optimisation of composites lay-up and combinations with the suitable matrix is to be made in order to increase the fibre/matrix interface bonding, which may increase the mechanical and the fatigue properties.

Acknowledgments

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